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Push-to-pull tensile testing of ultra-strong nanoscale ceramic–polymer composites made by additive manufacturing[☆]

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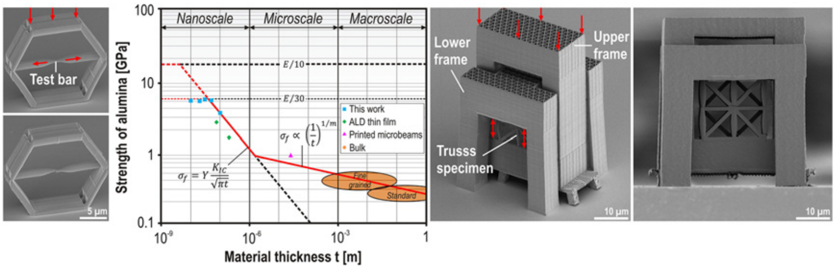
GRAPHICAL ABSTRACT



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ABSTRACT

The search for light yet strong materials recently benefited from novel high resolution 3D-printing technologies, which allow for fabricating lightweight porous materials with optimally designed micro-topologies. Architectural design improves mechanical properties significantly compared to stochastic porosity, as in foams. Miniaturization of the architectures offers to exploit material strengthening size-effects occurring at the nanoscale. However, these effects and their interaction with structural behavior are not yet well understood. We present tensile experiments of nanoscale alumina–polymer composite bars and cellular microarchitectures, applying 3D-printed push-to-pull mechanisms. The strength of alumina is found to strongly increase as the material thickness decreases. Below 50 nm thickness a plateau at about 5.5 GPa is reached, which is in the range of the theoretical strength. The characteristic low tensile strength of ceramics and its high variability seem not to hold at the nanoscale. Thus, when designed and fabricated appropriately, microarchitectures will facilitate carrying these size-effects beyond scales in future, allowing the use of ceramic materials far beyond what is possible to date.

☆ *Author Contribution*: J.B. and O.K designed research; J.B. and C.L. designed structures; J.B. and A.S. manufactured samples; J.B., A.S. and R.S. performed experiments; J.B. and I.T. performed finite-element simulations. J.B., O.K., L.V., R.S. and I.T. analyzed data; and J.B. wrote the paper .

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1. Introduction

Various examples of microarchitected 3D-printed cellular materials (Fig. 1(a)), aiming to combine both outstanding mechanical properties and low density, have been published recently [1–4]. Regardless of the length-scale of structuring, optimally designed architectures exhibit superior mechanical properties compared to stochastic cellular materials, such as technical foams [5,6]. However, whether or not microarchitecture provides any significant advantage over macroscopic topologies, with respect to the strength-to-weight ratio, depends on the impact of size-dependent material strengthening effects [7–9]. These occur at the sub-micrometer to nanometer scale, and exploiting them at the cellular-material level requires a careful design of the microarchitecture.

Assuming a perfect crystal, the theoretical strength, σ_{th}, of brittle materials, such as ceramics, is considered to be of the order of 1/10 of their Young's modulus, E [7,11]. However, failure generally occurs at considerably lower stresses, since all solid materials contain a variety of imperfections, ranging from lattice defects on the atomic level to voids and cracks on the micro- and macro-scale.

At the nanometer scale, mechanical size-effects have been shown to strongly enhance the strength of brittle materials [7]. Certain biological ceramic-like materials, such as enamel [12], nacre [13] or bone [14], exploit that effects by hierarchical structuring, with only a few nanometers thin plate-like basic building units. At small dimensions, it can be assumed that the flaw size scales with the size of the component, which for the case of a thin plate, would correspond to its thickness. Thus, the strength increases with decreasing size, and has been predicted to reach theoretical strength at a critical thickness [7]. Arranging a large volume fraction of nano-sized platelets in a small amount of organic matrix nature produces tough and strong materials at the macroscale [7].

It remains a challenge to maintain extreme size-dependent strengthening, as observed in controlled nanomechanical experiments [9] across length scales, to make it accessible for macroscopic applications, such as achieving high toughness and reasonable strength, as nature does [7]. Microarchitected cellular materials provide a very promising avenue though. High-resolution 3D-printing [15–17] of polymeric truss- or shell-structures in conjunction with conformal coating techniques such as atomic layer deposition [18] (ALD) allows manufacturing of micro-architected ceramic [2,3] or ceramic–polymer composite [1] structures, with typical dimensions of individual structural elements in the micrometer range. The ceramic shells need to be fabricated sufficiently thin to gain enhanced strength but still thick enough to prevent structural instability, particularly in case of hollow structures [2,3], or poor reinforcement in case of a composite design [1]. For engineering ceramics, it is expected that the thickness of the shells need to be reduced to the order of 10 nm to fully exploit size-dependent strengthening. Whether due to exceeding the resolution limit of the printing method [3] or processing related design limitations [4], buckling or beam bending, both undesired when aiming for high strength, may occur.

3D direct laser writing [15] (3D-DLW) enables fabrication of truss structures with sub-micrometer resolution, and high freedom of design. Lightweight microarchitectures with densities well below 1 g/cm^3 were recently shown to reach strength-to-weight ratios comparable to those of bulk structural materials under uniaxial compression [1] (Fig. 1(b)). Of course, the overall volume of a sample which may be manufactured by 3D-DLW within a reasonable amount of time is currently below 1 mm^3. Nonetheless, as the scalability of high-resolution additive manufacturing increases rapidly, comprehensive characterization techniques that allow investigating size-dependent material strengthening, structural behavior and their interaction under different loading conditions are required.

Up to now, microarchitected cellular materials have only been characterized under compression, which may be related to experimental difficulties such as handling micro-size test samples. Without the need to grip samples, a push-to-pull construction transforms an applied compressive loading into a tensile load on a specimen. This test method was originally developed in the mid-20th century [19] for macroscopic samples, but has not been used extensively, due to high machining effort and material consumption [20]. More recently, it was adapted to micro-scale experiments [20], significantly reducing experimental effort and error related to sample mounting and load application.

In this paper, we present an approach to push-to-pull tensile characterization of nanoscale ceramic–polymer composite tensile bars (Fig. 2) as well as three-dimensional microarchitectures (Fig. 5). Using 3D-DLW, test specimens were integrally manufactured with a push-to-pull construction in one single production step from polymer (IP-Dip). Structures were conformally coated with alumina (Al_2O_3) layers of 10–100 nm thickness, resulting in a ceramic–polymer composite. We performed uniaxial *in* and *ex-situ* tensile tests for mechanical characterization.

Our results indicate that below a certain thickness in the range of 50 nm, the tested alumina layers can be considered to be insensitive to flaws, reaching strengths of the order of the theoretical limit. Once the architectural length-scale of a structure is small enough to apply such thin walls appropriately, size-effects in ALD alumina can fully be translated into cellular microarchitected materials of much larger scale, which are superior to macroscopically architected cellular materials. Outperforming most foam materials [10], we demonstrate micro-truss structures with a density of 0.2 g/cm^3 for 50 nm thick alumina coatings which exhibit tensile strengths of up to 7 MPa.

2. Experimental approach and results

2.1. Tensile bars

Push-to-pull tensile bar experiments were performed by designing hexagonal frames with integrated horizontal test struts (Fig. 2(a)). The structures consist of a polymer core, printed by 3D-DLW, and a conformal ALD alumina coating with 10–100 nm thickness. By using a nanoindentation system equipped with a flat punch tip, the frames